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Journal of Sedimentary Petrology

A Publication of the Society of Economic Paleontologists and Mineralogists
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The American Association of Petroleum Geologists

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Journal of Sedimentary Petrology

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NOTICE

Articles submitted for publication to the JOURNAL OF SEDIMENTARY PETROLOGY should have a prefatory abstract and sufficient postage should be sent by the author to pay the return of the manuscript as it has been found that many articles require more or less revision before publication.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 5, NO. 3, FIG. 1, PP. 109-114, DECEMBER, 1935

FREQUENCY PERCENTAGE DETERMINATIONS OF DETRITAL QUARTZ AND FELDSPAR

R. DANA RUSSELL

Louisiana State University, Baton Rouge, Louisiana

ABSTRACT

Certain types of detrital quartz grains are described which may easily be confused with some of the plagioclase feldspars, particularly oligoclase and andesine. These grains appear to be quite common in some sediments. Their presence in any abundance slows down the accurate determination of frequency percentages of the minerals to a considerable extent. A simple staining method is described which eliminates this difficulty.

Quartz is so easily distinguished from the plagioclase feldspars in thin section that the possibility of confusing them does not occur to most petrographers. In detrital grains, however, this distinction may be quite troublesome. This is especially true when several hundred grains must be counted in order to determine percentages. In such cases, the time involved in securing interference figures on doubtful grains becomes an important factor.

Some of the feldspars can be readily distinguished from quartz on the basis of their indices of refraction. If the grains are mounted in Canada balsam or kollolith,[1] albite and the potash feldspars may be distinguished by their lower indices of refraction. The calcic members of the plagioclase group stand out with somewhat greater relief than quartz, but it is difficult to use this criterion in percentage determinations. Oligoclase and andesine, however, whose indices are fairly close to those of quartz, cannot be easily distinguished on the basis of relative indices. In many sediments these feldspars are the most common types present. If quartz always occurred without cleavage or parting, and the plagioclases always showed good cleavage, twinning, or both, distinction between them could still be made readily. Unfortunately this is not the case.

Feldspar crystals are commonly somewhat flattened, or tabular, parallel to 010. For this reason more than half of the grains of detrital feldspar are likely to lie on this face, even though the 001 cleavage is usually better developed than the 010. In these grains, lying on 010, the presence of twinning on the albite law is manifested only by imperfect extinction under crossed Nicols or,

[1] An artificial mounting medium made by Voigt and Hochgesang, Göttingen, Germany. It is colorless, amorphous, with an index of refraction of 1.5354, and has the advantage that it remains stable and does not decompose with time. It may be secured either in solid form or dissolved in xylol.

if a twin lamella projects past adjacent lamellæ, by slightly different extinction angles for the individual lamellæ. Even if the cleavage on such grains is evident, which it frequently is not, these grains may be easily confused with certain types of quartz, unless their optical character is determined by means of an interference figure.

There are several types of quartz grains which greatly resemble grains of detrital plagioclase feldspar.

(1) Rounded or irregularly shaped grains with imperfect extinction. Such grains may be confused with rounded or irregularly shaped plagioclase grains, in which the cleavage is not evident, which are lying parallel or nearly parallel to the 010 plane.

(2) Grains lying upon a comparatively flat surface, resembling a cleavage or crystal face, and showing imperfect extinction. Such grains resemble plagioclase grains lying upon the 010 cleavage or crystal face, with the 001 cleavage not evident.

(3) Grains lying upon a comparatively flat surface, resembling a cleavage or crystal face, with the development of a plane of fracture (parting or cleavage) nearly at right angles to the surface upon which the grains are lying, and showing imperfect extinction.

A rather large percentage of these quartz grains, especially those of groups two and three, are composed of thin parallel or subparallel lamellæ of slightly different optical orientation. When these lamellæ project unequally at the edges of the grain, they show slightly different extinction positions. The effect is strikingly like that produced by the unequal projection of twinning lamellæ in plagioclase grains.

The causes of these "anomalies" in quartz are so numerous that a detailed discussion of them lies outside the scope of this paper. Only a brief review of the most important causes will be attempted.

(1) Imperfect extinction may be caused either by strain (the so-called "undulatory extinction," "wavy extinction," or "strain shadows" commonly shown by quartz and other minerals in metamorphic rocks) or by the superposition of platy elements of slightly different optical orientation.[2]

(2) "Platy quartz," consisting of lamellæ oriented in a parallel or subparallel manner, may be the result of:

(a) Normal crystallization, in which thin plates of slightly different orientation grow side by side. Adams[3] states that the feathery and flamboyant quartz of veins is of this mode of origin.

(b) The replacement of a platy mineral by quartz.[4]

(c) The shearing of quartz crystals.

(d) The recrystallization of quartz when a planar control is involved in the recrystallization.

[2] The phenomenon of rotary polarization in quartz also produces imperfect extinction, but only in fairly thick grains whose plane of rest is perpendicular, or nearly perpendicular, to the optic axis.

[3] Adams, S. F., A microscopic study of vein quartz: *Econ. Geol.*, vol. 15, pp. 623–664, 1920.

[4] Adams, S. F., *Op. cit.*, p. 644.

(e) The development of secondary lamellar twinning.[5]

(3) The parting or cleavage observed on many of the grains may be the rhombohedral cleavage sometimes shown by quartz, or a parting induced by strain.[6] The writer has observed that when the parting or cleavage is particularly well developed, the orientation of the flat surface, as shown by the degree off-center of the interference figure yielded by grains lying upon the surface, is approximately parallel to the unit rhombohedron. Apparently a cleavage or parting in quartz is much commoner than has been supposed.[7]

Quartz of the various types described above appears to be rather common in sediments but has not received much attention, perhaps because all of the light minerals have been somewhat neglected in favor of the more interesting heavy minerals.[8] This attitude is probably a mistake, since a study of the lighter minerals often yields more information than can be derived from a study of the "heavies."

Recent studies show that quartz of all three types is very common in sands from the bed of the Mississippi River. In many samples, more than half of the quartz grains show imperfect extinction with the frequent development of one plane of parting or cleavage and occasionally of two or three. Platy structure is also common. A few grains showing two or three planes of parting, with well developed platy structure, are shown in the photomicrograph (fig. 1). In addition, more than half of the plagioclase feldspar grains lie upon 010 and in many of them the cleavage is not evident, so that the number of doubtful grains is rather great. In such cases, the determination of frequency percentages of the minerals is slowed down tremendously by changing objectives and securing interference figures on the doubtful grains.

The time involved in frequency determinations can be greatly reduced by the use of a staining method which will afford an immediate sep-

[5] According to Judd (*Mineralogical Mag.*, vol. 8, p. 1, 1888) lamellar twinning, parallel to the positive or negative rhombohedron, may be developed in quartz by pressure.

[6] Most textbooks of mineralogy refer to these planes of separation in quartz as rare or imperfect cleavage, but Dana (*A Textbook of Mineralogy*, 4th ed., John Wiley and Sons, New York, 1932) describes them as fracture surfaces. Lehmann (*Zeitschrift für Krystallographie und Mineralogie*, vol. 11, p. 608, 1886) and Judd (*op. cit.*, p. 7) regard them as gliding planes. They may be developed in a crystal, which does not ordinarily show them, by heating the crystal and then plunging it into cold water. F. D. Adams (*Jour. Geol.*, vol. 18, p. 510, 1910) produced similar planes of separation in quartz by subjecting a crystal to pressure.

It seems probable that processes of weathering, particularly the expansion and contraction resulting from temperature changes, may develop this feature in quartz grains.

[7] A. F. Rogers has recently emphasized the prevalence of this feature. (*Am. Mineralogist*, vol. 18, p. 111, 1933; Abstract).

[8] The classic work of Mackie on the sands and sandstones of Eastern Moray (*Edinburgh Geol. Soc. Trans.*, vol. 7, p. 159, 1899) is an exception. Mackie mentions "the fine parallel cleavage of quartz, often in more than one direction in the same grain," "polysynthetic quartz . . . showing iron staining in the interstices," and "dark turbid quartz with parallel joint lines." These grains were noted in the lower Old Red sandstone. Describing samples from the upper Old Red sandstone, he mentions the "peculiar wavy cleavage of some quartz grains." He considered the source to be a granite in each case.

aration of the quartz from the other minerals. Gabriel and Cox[9] have described a method which consists in subjecting the dry material to hydrofluoric acid fumes and then staining it with sodium cobaltinitrite solution. After treatment, the potash feldspar grains are intensely yellow, quartz is transparent, and the plagioclases are white and opaque. If desired, the material may be treated a second time, using eosine, which stains the plagioclase feldspars, but leaves the potash feldspars predominantly yellow.

FIG. 1.—Photomicrograph (×81) of detrital quartz grains showing cleavage or parting. The grains are lying upon one cleavage or parting plane with a second and, in most cases, a third shown by the outline or by lines of fracture. Platy structure, most evident under crossed Nicols, is well developed in all of the grains.

If no further division of the feldspars is desired, this method is quite satisfactory, but more detailed determination of the feldspars would require the examination of the unstained material as well. Unfortunately, this is true of any staining method yet devised. The method also has the disadvantage that it requires a special lead box apparatus.

Determination of the mineral percentages in a rather large number of samples from the bed of the Mississippi River necessitated a simple, accurate, and rapid staining method. After some experimentation, a method was devised which fulfilled these requirements, and for which no special apparatus is needed. The procedure is as follows:

The sample to be analyzed is cut down in a sample splitter[10] to twice the amount necessary for mounting on a microscope slide. This is then cut in half, and one-half prepared as an ordinary slide mounted in kollolith. The other half is used for staining. A slide glass is covered with a film of kollolith whose thickness is equal to about half the diameter of the grains to be examined. After this film has hardened, the grains are distributed uniformly over the surface of the kollolith, covering an area the size of a cover glass. The slide is then

[9] Gabriel, A., and Cox, E. P., A staining method for the quantitative determination of certain rock minerals: *Am. Mineralogist*, vol. 14, pp. 290–292, 1929.

[10] The "microsplit," designed by G. H. Otto, and described in vol. 3, p. 30, of this Journal, is used for this purpose and gives very satisfactory results.

heated until the grains sink down into the softened kollolith, leaving their upper portions exposed. After cooling, the grains are covered with a drop or two of hydrofluoric acid, which is allowed to remain on for about one minute and is then washed off. The acid produces a gelatinous film of aluminum fluosilicate on the feldspars and other aluminous minerals which may be present, but leaves the quartz clear. The acid must be washed off gently, otherwise the film on the feldspar grains will be removed. After washing, the grains are covered with a water-soluble organic dye for about five minutes, and again washed. Of the several dyes tried for this purpose, malachite green was found to give the most consistently good results. After drying, the slide may be mounted with kollolith if a permanent mount is desired, or, for routine examination, covered with an oil of the same index of refraction as the kollolith. The frequency percentage of quartz is then determined by making several traverses across the slide by means of a mechanical stage, counting the grains of quartz, and of all other minerals grouped together. At least 300 grains are counted, usually between 400 and 600.

Frequency percentages of the other minerals are counted in the same manner from the untreated slide, quartz and plagioclase grains more calcic than albite being grouped together. From the total percentage of quartz plus these plagioclases, the percentage of quartz as determined from the stained slide is subtracted, the remainder being the percentage of the plagioclases other than albite. In practice it is desirable to mount a third slide in an oil with an index of refraction of 1.560, for determination of the plagioclases more calcic than andesine. In sands from the Mississippi River, these plagioclases make up less than one per cent of the sample. The final determination thus gives the percentages of microcline, orthoclase, sanidine, albite, oligoclase and andesine (grouped together), and the plagioclases more calcic than andesine, in addition to the percentage of quartz and of other minerals.

This method may be criticized for two reasons. First, the splitting of the sample introduces the possibility of percentage errors in the final result. A sample will rarely be divided in such a manner that the two halves contain exactly the same percentages of each mineral. Since the final percentages are derived from two slides (disregarding the slide checked for the presence of labradorite, bytownite, and anorthite), an error in random distribution in either slide will produce an error in the final result. To determine the magnitude of this error a check of the method was made by determining percentages in duplicate stained slides from the same sample. About fifty samples were thus examined. In almost every case, the difference between the quartz percentages in the duplicate slides was less than three per cent in a total quartz percentage of 55 to 70. Nearly half of the duplicate

slides checked exactly (percentages were determined only to the closest whole per cent). A series of apparently identical samples collected from the same locality would show a greater variation in percentages than this.

The second reason for criticism is the time involved in the preparation and counting of the stained slides. With practice, these may be prepared at the rate of about four an hour. Each slide may be counted, with the aid of a mechanical tally, in 15 to 20 minutes. Thus 30 to 35 minutes per slide is required in addition to the time taken to determine percentages in the unstained slide. This, however, is very much less than the time needed, in ordinary slides, to secure interference figures on the doubtful grains, even if the latter make up no more than fifteen per cent of the sample. Experience has shown that the use of this method not only saves time, but also results in greater accuracy.

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DECEMBER, 1935

INSOLUBLE RESIDUES FROM PALEOZOIC LIMESTONES OF THE MOSQUITO RANGE, COLORADO[1]

QUENTIN D. SINGEWALD AND CHARLES M. REED
University of Rochester, Rochester, New York

ABSTRACT

The general characteristics of insoluble residues from five dolomitic limestone units, in the Paleozoic formations of the Mosquito Range, are described. In general, isolated residues from these formations have little diagnostic value, but groups of residues from several beds within a narrow stratigraphic range have distinctive features that furnish a valuable and readily utilized aid in correlation.

INTRODUCTION

The value of insoluble residues of limestones as an aid in stratigraphic correlation has been shown by H. S. McQueen[2] and his associates of the Missouri Bureau of Geology and Mines. The senior author became especially interested in this work when, in 1933, during Excursion A-2 of the International Geologic Congress, he had an opportunity to examine some residues prepared by McQueen. As a result, it was decided to apply the technique to a study of the Paleozoic limestones of the Mosquito Range of central Colorado. Only twenty-four specimens from various outcrops within an area of about 45 square miles were available, so the study is far from exhaustive. Nevertheless, certain results are sufficiently definite to be worth publishing, since they may be utilized advantageously by anyone prospecting, especially by diamond drilling, for metallic deposits in the region. The results have only indirect interest to petroleum geologists, as none of the formations produce oil.

Field work in connection with this paper was done by the senior author in the course of a study of the geology and ore deposits of the Alma district, a part of the cooperative work, under the direction of B. S. Butler of the U. S. Geological Survey, the State of Colorado, and the Colorado Metal Mining Fund. Most of the laboratory work was done by the junior author, at the University of Rochester. The laboratory technique was nearly as outlined by McQueen. A little more than 10 grams of each hand specimen was broken and sieved. The portion ranging from one-tenth of an inch to one-fourth of an inch in diameter then was weighed and treated with 50 cubic centimeters of dilute C.P. hydrochloric acid. As all the lime-

[1] Published with the permission of the Director of the United States Geological Survey.

[2] McQueen, H. S., *Insoluble residues as a guide in stratigraphic studies: Missouri Bur. Geol. and Mines, 56th Bien. Rept.*, Appendix 1, 1931.

stones are dolomitic, the samples were placed on a sand bath during the first two or three hours. The insoluble residues were dried, weighed, and studied under both binocular and petrographic microscopes. In this paper, the descriptions of the residues include only general characteristics that may be determined fairly rapidly, for great detail on the basis of elaborate and time-consuming study would vitiate the very purpose desired.

GENERAL STRATIGRAPHY

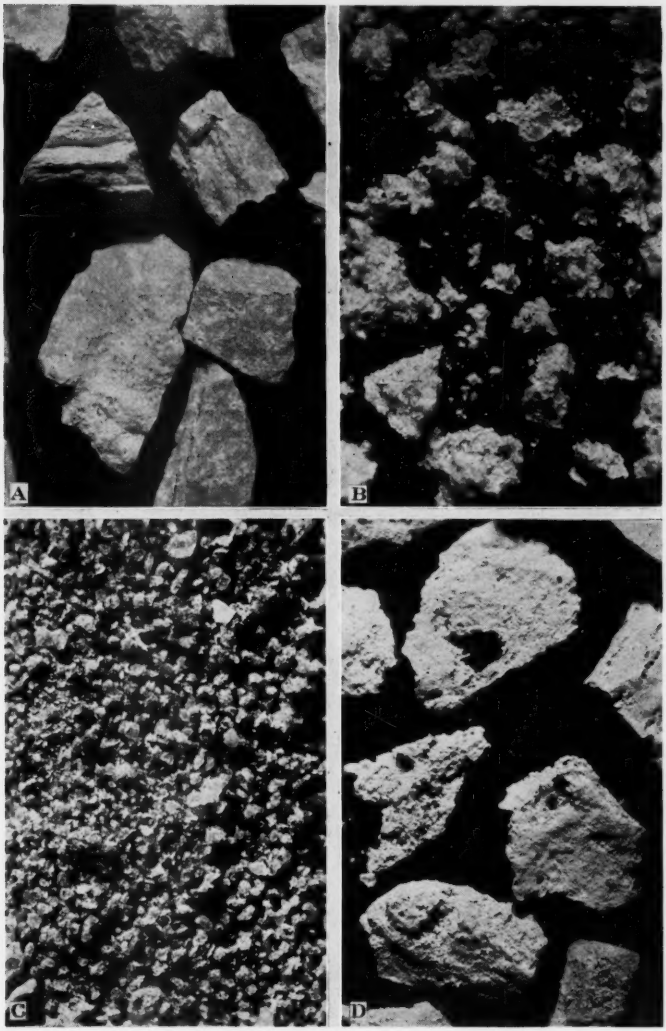
All the specimens studied came from the northeastern part of the Mosquito Range, located northwest of South Park in central Colorado. Complete stratigraphic detail for this region may be obtained from several publications.[3] The accompanying chart of the stratigraphic column gives the thickness of the formations and zones and other information pertinent to this paper. Special attention may be called to the erosional un-

[3] Behre, C. H., Jr., Revision of structure and stratigraphy in the Mosquito Range and the Leadville district, Colo.: *Colo. Sci. Soc., Proc.*, vol. 12, pp. 38–42, 1929. Emmons, S. F., Irving, J. D., and Loughlin, G. F., Geology and ore deposits of the Leadville mining district, Colo.: *U. S. Geol. Survey, Prf. Paper* 148, pp. 25–42, 1927. Singewald, Q. D., and Butler, B. S., Preliminary geologic map of the Alma mining district, Colo.: *Colo. Sci. Soc., Proc.*, vol. 12, no. 9, pp. 295–302, 1930.

GENERAL STRATIGRAPHIC COLUMN OF NORTHERN PART OF THE MOSQUITO RANGE, COLORADO

AGE	SUBDIVISIONS (AS ADOPTED BY COMMITTEE ON GEOLOGIC NAMES)	LITHOLOGIC ZONE	THICKNESS	DESCRIPTION	TYPICAL INSOLUBLE RESIDUES
PENNSYLVANIAN	WEBER (?) FORMATION		1800?	INTERBEDDED QUARTZITE, CONGLOMERATE, GRIT, ARKOSE, SHALE AND OCCASIONAL LIMESTONE, ALL MICACEOUS. SOME SHALE HIGHLY CARBONACEOUS. SHALE PREDOMINATES NEAR BASE, SANDY BEDS IN UPPER PART	LIMESTONE BEDS YIELD RED OR GREEN HARD, ANGULAR FRAGMENTS.
	— HIATUS —				
MISSISSIPPIAN	LEADVILLE LIMESTONE	LIMESTONE ZONE	0–170'	BLUE TO BLACK, MOSTLY DENSE-TEXTURED, MASSIVE-BEDDED DOLOMITIC LIMESTONE. SHATTERS EASILY. WEATHERS WITH PITTED SURFACES AND BREAKS INTO BLOCKY FRAGMENTS. "ZEBRA ROCK," CHERT, AND LIMESTONE BRECCIA ARE COMMON.	VERY DARK GRAY, SHALY POWDER
	— HIATUS —				
DEVONIAN	CHAFFEE FORMATION — DYER DOLOMITE MEMBER	QUARTZITE ZONE	0–8'	FINE-GRAINED TO DENSE "CHERTY-LOOKING" WHITE QUARTZITE. LENTICULAR.	
			40'–80'	FAIRLY THIN-BEDDED, MOSTLY DENSE WHITE AND BLUE DOLOMITIC LIMESTONE. WHITE BEDS WEATHERED CREAM-COLORED. EXPOSED SURFACES GENERALLY SMOOTH.	LIGHT COLORED SHALY POWDER WITH A FEW LARGER FRAGMENTS.
	— PARTING QUARTZITE MEMBER		0–35'	CROSS-BEDDED AND CONGLOMERATIC QUARTZITE AND SANDY LIMESTONE. QUARTZ PEBBLES SUBANGULAR. LOCALLY SLIGHTLY SHALY. WEATHERS BROWNISH GRAY.	
	— HIATUS —				
ORDOVICIAN	MANITOU LIMESTONE		0–130'	THIN-BEDDED WHITE AND MEDIUM BLUE, MOSTLY CRYSTALLINE DOLOMITIC LIMESTONE. WEATHERS LIGHT GRAY DEVELOPING SILICEOUS RIBBING. BREAKS TO SLABBY FRAGMENTS. LOCALLY, SLIGHTLY SHALY AT TOP.	LIGHT COLORED, RANGING FROM EARTHY CHERT FRAGMENTS TO HACKLY POWDER.
CAMBRIAN	SAWATCH QUARTZITE — PEERLESS SHALE MEMBER	UPPER ZONE	30'–55'	INTERBEDDED DOLOMITIC LIMESTONE, SHALY LIMESTONE AND LIMY SHALE. LIMESTONE PREDOMINATES. SHALE WEATHERS GREEN LIMESTONE DRAB TO BROWN	RANGE FROM LIGHT COLORED, POROUS SHALE FRAGMENTS TO HACKLY POWDER.
		MIDDLE ZONE ("TRANSITION SHALE")	35'–65'	THIN-BEDDED, ALMOST FLAGGY SERIES OF DOLOMITIC LIMESTONE AND SILICEOUS SHALE. SHALE WEATHERS GREEN. LIMESTONE BROWN. RED CASTS IN UPPER LIMESTONES.	LIGHT COLORED RANGING FROM SLIGHTLY POROUS SHALE FRAGMENTS TO VERY HACKLY POWDER.
		PURPLE QUARTZITE ZONE	2'–15'	PURPLE TO BLACK, FAINTLY CONGLOMERATIC AND CROSS-BEDDED QUARTZITE.	
	— HIATUS(?)		60'–130'	FAIRLY THICK-BEDDED, WHITE, FINE-GRAINED QUARTZITE. CONGLOMERATE AT BASE. CALCAREOUS QUARTZITE INTERBEDDED IN MIDDLE PART.	
PRE-CAMBRIAN				GNEISSES, SCHISTS, GRANITES.	

General stratigraphic column of the northern part of the Mosquito Range, Colorado.



EXPLANATION OF PLATE 1

Fig. A, Whitish-gray, fairly hard, rough shale fragments; residue from a limy shale bed in the middle zone of the Peerless, ×6.

B, White, very coarse and hackly, shaly powder; residue from a limy bed in the middle zone of the Peerless, ×6.

C, Slightly greenish, medium gray, moderately coarse and hackly, shaly powder, residue from a limy bed in the upper zone of the Peerless, ×6.

D, Light-gray, cavernous, earthy-looking chert fragments; residue from the Manitou, ×6.

conformities at the base of the Parting quartzite, the Leadville limestone, and the Weber (?) formation, all of which cause variations in the thickness of and locally the complete absence of the underlying formation. The one at the base of the Weber (?) formation is the most important. Fossils are entirely absent in all of the formations at the vast majority of outcrops.

PEERLESS SHALE

The Peerless shale member of the Sawatch quartzite, as shown in the stratigraphic column, is here divided into three zones. The lower zone is a purple quartzite that grades upward into the middle zone.

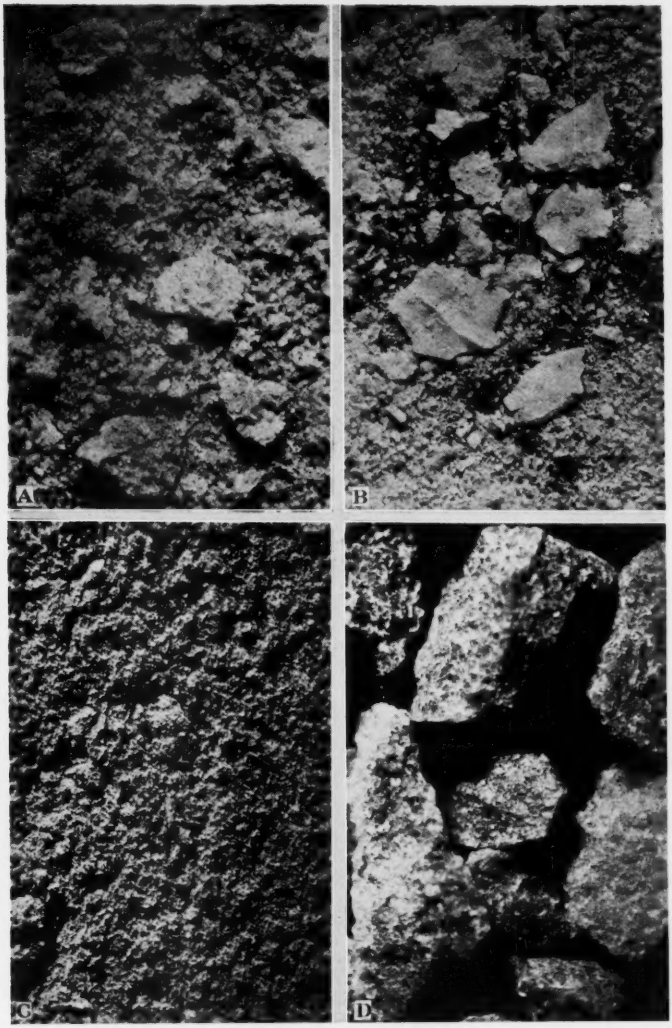
The middle zone is a thin-bedded series of siliceous shale, shaly limestone, and limestone. The limestone beds, all of which are dolomitic, are abundant near the base but decrease in abundance upward; those near the top contain numerous "red casts."[4] The shales contain very little soluble material, but as the limy content increases the insoluble residue decreases to a minimum of 15 per cent (by weight). The residues are white, whitish-gray, or very pale-pink; and they range, according to the original limy content, from fairly hard, rough fragments having irregularly distributed tiny pores, to exceedingly fragile and spongy material that is largely broken down to a coarse, hackly powder. The residues are predominantly shale, though some contain a considerable quantity of small, frosted quartz grains. A rather shiny luster, due to reflection of light from innumerable minute sericite flakes is characteristic. Sericite, pale chlorite, quartz, and clay minerals are the chief constituents other than carbonates seen in thin sections.

The upper zone is decidedly more limy and less thinly bedded than the middle zone, but in other respects the two zones are lithologically similar. Like the middle zone, the upper zone becomes increasingly shaly toward the top. Insoluble residues from the limestones of this zone are darker and smaller in quantity than those of the middle zone. Typical ones are slightly greenish, medium gray shaly material, consisting of exceedingly fragile and spongy fragments and a rather coarse, hackly powder. The chief constituents are sericite, pale chlorite, and clay minerals. One residue, however, is a slightly reddish, medium gray sand, composed of frosted quartz and feldspar poorly cemented by shaly constituents. The shaly beds of the upper zone yield light-gray, fairly hard, rough and porous fragments almost identical with those from similar beds in the middle zone.

MANITOU LIMESTONE

The Manitou limestone differs from the Peerless shale in color, in development of a siliceous ribbing on a weathered surface, and in the almost complete absence of shale. The insoluble residues, comprising from 8 to 30 per cent of the rock, are white to light-gray. Some consist entirely of fairly soft, earthy-looking

[4] Emmons, S. F., Irving, J. D., and Loughlin, G. F., *op. cit.*, pl. 36-B.



EXPLANATION OF PLATE 2

Fig. A, Light-gray, moderately coarse and hackly, shaly powder with small fragments; residue from the Manitou, ×6.
B, White, shaly powder with small flaky fragments; residue from the Dyer, ×6.
C, Dark-gray, shaly powder; residue from the Leadville, ×6.
D, Pale-red, angular, sandstone fragments; residue from a limy bed of the Weber (?) formation, ×5.

chert fragments having cavernous pores of diverse size, shape, and arrangement; others consist of an earthy-looking, slightly hackly powder with almost no larger fragments; and gradations exist between these two extreme types. The larger fragments, as seen under a petrographic microscope, have minute grains of quartz and feldspar and matted flakes of sericite and clay minerals sparingly distributed in a partly isotropic, partly crypto-crystalline siliceous matrix. As the quantity of siliceous matrix decreases, the residue becomes more powdery. Under a hand lens or a binocular microscope the residues of the Manitou limestones are not so readily distinguished from residues of limy beds in the middle zone of the Peerless shale. The Manitou residues, of course, do lack the slightly porous shale fragments characteristic of the shaly beds of the Peerless.

DYER DOLOMITE

Almost everywhere the Dyer dolomite member of the Chaffee formation is separated from the Manitou limestone by the Parting quartzite member of the Chaffee, an easily recognized division, but at a few places the Parting quartzite is absent.[5] The general absence of siliceous ribbing, the creamy color, the dense texture, and the smooth and soft-looking surfaces serve readily to distinguish a weathered outcrop of the Dyer from the Manitou. Typical insoluble residues, comprising from 2 to 12 per cent of the rock, consist of a white or whitish-gray, somewhat hackly powder of shaly material with a few very spongy fragments. Powder and fragments consist of sericite and clay minerals, with subordinate amounts of very fine-grained quartz. Hence, the residues under a binocular microscope resemble in general appearance the more powdery residues of the Manitou limestone. At a few places, the lowest beds of the Dyer dolomite contain angular quartz pebbles, and so yield a white sandy residue.

LEADVILLE LIMESTONE

The Leadville limestone contains much "zebra rock"[6] and is darker colored, contains more chert nodules, is more massively bedded, and weathers to rougher surfaces than the Dyer dolomite, yet in spite of these differences the two formations in places are difficult to distinguish from each other. The insoluble residues of the Leadville limestone, however, are distinctive. Those from specimens free of chert are very small in amount, comprising from less than one-half of 1 per cent to a maximum of 5 per cent of the rock, and consist of a dark-gray, slightly hackly, shaly powder. The dark color is due to an abundance of carbonaceous material. The chief mineral constituents are clay minerals, very fine-grained quartz, and sericite.

WEBER (?) FORMATION

The Weber (?) formation consists mainly of clastic material and, there-

[5] Singewald, Q. D., Depositional features of the "Parting" quartzite near Alma, Colo.: *Am. Jour. Sci.*, (5), vol. 22, pp. 404–413, 1931.

[6] Behre, C. H., Jr., *op. cit.*, p. 41.

fore, is so easily distinguished from the underlying rocks that preparation of insoluble residues from the few interbedded dolomitic limestones is superfluous for correlation. Nevertheless, as a matter of interest in connection with this study, residues were prepared from two of the limestones. Both residues consist of hard angular fragments in which rare small flakes of muscovite are visible to the naked eye. The fragments of the one residue are pale red sandstone, of the other pale green shaly sandstone.

CONCLUSIONS

The insoluble residues from the calcareous formations below he Weber (?) beds are all more or less shaly. Moreover, the residues derived from beds of different lithology in the same formation or zone in certain instances differ more markedly from one another than residues from beds of approximately the same lithology in different formations. Consequently examination of one or two residues alone is generally not sufficient to determine stratigraphic position. On the other hand, a group of residues derived from different beds within a narrow stratigraphic range show either similarities or variations that are fairly characteristic of a particular formation or zone, and are therefore a valuable aid in distinguishing between adjacent units if the approximate stratigraphic position is known by other criteria such as distance above or below some easily recognized horizon marker. Some of the possible applications are given in the following paragraphs.

The middle and upper zones of the Peerless shale, because of their general lithologic similarity, are often difficult to distinguish in the field, but the group characteristics of the insoluble residues from each zone are fairly distinctive. All the residues from the middle zone are light-colored, whereas some from the upper zone are medium gray with a slightly greenish tone.

The Manitou limestone, on the other hand, is more readily distinguished from the Peerless shale by means of lithologic differences than by residues. However, small differences (as the silica in the Manitou residues), exist between groups of residues from these formations.

The Dyer dolomite, likewise, weathers quite differently than the Manitou limestone, and, moreover, the easily recognized Parting quartzite is almost everywhere present between them, so that insoluble residues usually are not necessary to distinguish between these formations. Slight differences between groups of residues from them, however, may be utilized where the correlation is in doubt.

The Leadville limestone and the Dyer dolomite, in spite of certain general lithologic differences, are difficult to distinguish from each other at many places, and either formation may directly underlie the Weber (?) beds according to the amount of pre-Weber (?) erosion at the particular locality concerned. Insoluble residues, however, furnish a ready means of identification, for the very dark color and small quan-

tity of residues from the Leadville limestone are quite characteristic. This separation has an important economic application, for the chances of finding rich silver-lead deposits beneath the Weber (?)' formation are much greater in the Leadville limestone than in any other formation.

In closing, it may again be emphasized that insoluble residues in this region have much value in correlation, but their function must be that of an aid rather than a main basis.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 5, NO. 3, PP. 123–132, FIGS. 1–3,
DECEMBER, 1935

THE INSOLUBLE RESIDUES OF THE CAMBRO-ORDOVICIAN LIMESTONES OF THE LEHIGH VALLEY, PENNSYLVANIA

JOHN M. HILLS
Amerada Petroleum Corporation, Midland, Texas

ABSTRACT

This paper is a short description of the insoluble residues obtained from the Cambro-Ordovician magnesian limestones of the Lehigh Valley, Pennsylvania. The methods of collecting the samples and preparing the residues are briefly described. The residues thus obtained are composed of shale, sand, dolocasts, and chert, with tests of arenaceous foraminifera at certain horizons, and almost no heavy minerals. By plotting the proportions of the various constituents in the residues it was found possible to establish certain definite stratigraphic horizons in the series. The foraminifera were especially useful in this work and the zones thus established were used to work out the structure and stratigraphy of the area. A table is given showing the formations differentiated by this method.

The formations dealt with in this paper form a section of magnesian to dolomitic Cambro-Ordovician limestones about 3,500 feet thick. They rest on an arkosic quartzite of Lower to Middle Cambrian age and are overlain by argillaceous limestones and shales of the Middle and Upper Ordovician. The details of thickness and character of the formations are shown in Fig. 1. The area studied lies near the junction of the Lehigh and Delaware rivers in eastern Pennsylvania, and forms part of the Appalachian Valley geological province. Reconnaissance sampling of formations of related age also was done in the Reading and New Holland quadrangles, which form part of the same geological province farther to the southwest.

The lithology of all parts of these limestones is very similar, and, while the upper portion of the series contains some Ordovician fossils which definitely fix its age as Beekmantown, the rest is practically unfossiliferous except for abundant *Cryptozoon* (*proliferum ?*). These and a few other fossils reported by Kummel and Weller,[1] indicate an Upper Cambrian age for the lower part of the section.

At the time the writer undertook this study the section had been only very roughly subdivided into formations of very doubtful internal succession. Since the structure of the region is very complex, the problem was to find a method whereby the stratigraphic succession of these intensely folded and faulted formations could be determined and used to unravel the structure of the area. The purpose of this paper is to tell why the method of insoluble residues was chosen, to give some account of the methods used to obtain the res-

[1] Kummel, H. B., and Weller, Stuart, Paleozoic limestones of the Kittatinny Valley, New Jersey: *Geol. Soc. America, Bull.*, vol. 12, pp. 147–164, 1904.

STRATIGRAPHIC COLUMN
LEHIGH VALLEY, PA.

THICKNESS	SYSTEM	NAME	LITHOLOGY	RESIDUES
200–400	ORDOVICIAN	JACKSONBURG LIMESTONE	Argillaceous thin-bedded limestone locally known as cement rock	
1000	ORDOVICIAN	UPPER BEEKMANTOWN LIMESTONE	Dark thin-bedded limestone Many thin shale beds Few cherty beds in middle Many high calcium beds	Largely shale, some sand Large dolocasts common Chert abundant above middle Many samples effervesce strongly
	ORDOVICIAN	LOWER BEEKMANTOWN LIMESTONE	Dark thin-bedded limestone with many shaly interbeds *Some high calcium beds, much mottling* Weathers laminated and gnarly	Much shale, little sand and chert
600	ORDOVICIAN	UPPER ALLENTOWN LIMESTONE	Massive dolomite beds with shaly interbeds becoming more abundant toward top. Mottling observed in some sections	Sand with equal amount of shale Some chert with a few dolocasts
	ORDOVICIAN	UPPER ALLENTOWN LIMESTONE	Largely thin-bedded dolomites and shaly limestones Some dark and light chert *Few poor cryptozoon beds*	Shale with slightly smaller quantity of sand Some chert *Few foraminiferal pockets*
700–800	CAMBRIAN	LOWER ALLENTOWN LIMESTONE	Dark magnesian limestone *Top of abundant cryptozoon zone*	Largely sand. Persistent foraminiferal zone
	CAMBRIAN	LOWER ALLENTOWN LIMESTONE	Dark magnesian limestone, some shaly Few shaly beds, Many cryptozoa *Conglomerate & crossbedded beds*	Shale in bottom samples becoming sandy toward top
	CAMBRIAN	LOWER ALLENTOWN LIMESTONE	Thin-bedded dolomites & shaly limestone Much thin pebble conglomerate Many well developed cryptozoon reefs	Much sand, some shale Several pockets of foraminifera toward top
	CAMBRIAN	LOWER ALLENTOWN LIMESTONE	Dark massive to thin-bedded dolomite Little flat pebble conglomerate, many Cryptozoa	Equal quantities shale and sand Few pockets of foraminifera
	CAMBRIAN	LOWER ALLENTOWN LIMESTONE	Shaly & sandy dolomite thin-bedded, much conglomerate & breccia. Many cryptozoa & oolite beds	Sandy with foraminifera. Top & bottom Berrain shaly zone in middle
450–650	CAMBRIAN	UPPER LEITHSVILLE FORMATION	Massive magnesian limestone beds 6'-8' thick alternating with shaly beds *Few cryptozoon & oolite beds in top* Becomes thinner bedded toward top	Mostly shale with several sandy zones Some dark chert but residues more often light colored. More sand in middle of zone. Few samples effervesce
	CAMBRIAN	UPPER LEITHSVILLE FORMATION	Dark massive magnesian limestone 6'-10' thick alternating with thinner shaly zones	Largely dark shale, few sandy samples. Some chert-one or two pockets foraminifera
	CAMBRIAN	UPPER LEITHSVILLE FORMATION	Sandy limestone, very large rounded grains. Weathers to porous quartzitic sandstone	Very large quantity rounded quartz grains much secondary enlargement
400–500	CAMBRIAN	LOWER LEITHSVILLE FORMATION	Massive dolomite interbedded with calcareous shales	Dark shale with small quantities of sand and chert from limestone beds
20–300	CAMBRIAN	HARDYSTON QUARTZITE	White to purple grits becoming locally arkosic to jaspery	

FIG. 1.

idues, and to describe briefly their chief characters. The location and geologic situation of the areas studied is shown on the map (Fig. 2).

METHODS OF STUDY

Field.—Different methods to determine the stratigraphic sequence and correlate the various exposures found in the field were considered and tried, but no method of direct comparison of the outcrop characters gave satisfactory results. This is due largely to the fact that the heterogeneity of the beds and the complexity of structure make it impossible to be certain that exposures which have even a small gap between them are stratigraphically continuous. A short trial of the method of insoluble residues was made in the fall of 1930. The results were sufficiently encouraging so that the field seasons of 1932, '33, and '34 were devoted to collecting samples from which residues were made and studied during the winter. Some 950 samples were collected which included representatives of nearly all important exposures in the Lehigh and Delaware valleys. The first 500 samples taken during the 1932 field season were spot samples of some two pounds apiece, taken at intervals of about 50 feet stratigraphically. Those collected during the next field seasons approximated channel samples, each of which, covering about 20 feet stratigraphically, weighed a pound to a pound and one-half. This method of sampling was made possible only by the fact that the limestones are extensively exposed along railroads and highways following the river trenches where they generally dip at fairly steep angles.

The location of each sample was referred to bench marks which were cut into the exposures and carefully located on the map. The dip and strike were noted at frequent intervals, especially where the attitude of the beds changed very rapidly. Sketches were made of exposures where particularly interesting or complex structures were seen, and the location of the samples was marked on the sketches so that their relative positions could be readily determined.

Laboratory.—The technique of treating the samples differed in several respects from that of McQueen[2] and therefore will be described here in some detail. Chips were broken from each piece of the field sample, care being taken to have each variety of limestone in the sample fairly represented in the chips. These were reduced with a hammer to a maximum diameter of one-half inch, and then quartered down to a laboratory sample of 50 grams. This large quantity was used because some of the limestones contained so little insoluble material that a small sample yielded scarcely enough residue for study. On the other hand, a large sample was inconvenient in the case of some of the very impure beds where the residue was upwards of two-thirds of the limestone, so that

[2] McQueen, H. S., Insoluble residues as a guide in stratigraphic studies: *Missouri Bur. Geol. and Mines, 50th Bien. Rept.*, Appendix 1, pp. 104–107, 1931.

large four-dram vials had to be used for storage of the residues.

The samples were placed in ordinary ten-ounce water glasses and dissolved with about 100 cc. of cold commercial hydrochloric acid to which 100 cc. of water were added.

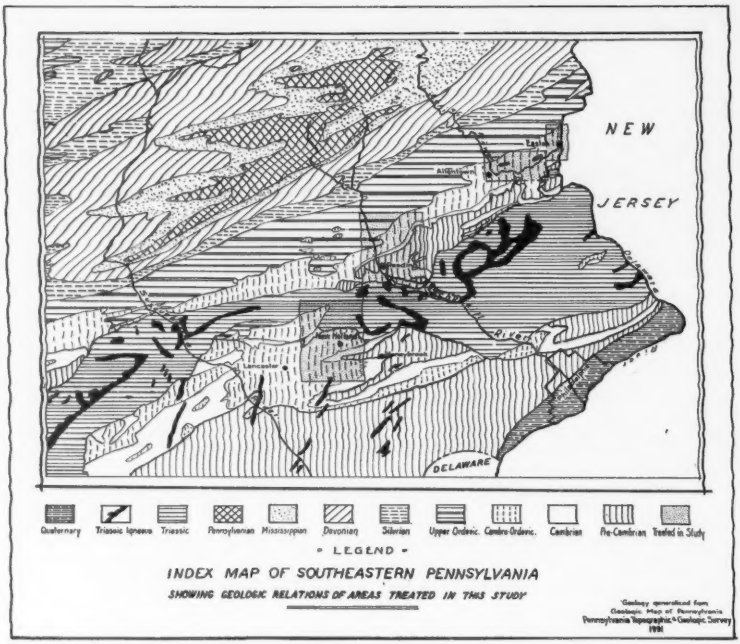

FIG. 2.

The dilution of the acid was carried out in the presence of the limestone so as to take advantage of the heat of solution in hastening the reaction with the sample. They were left to digest for about 48 hours with occasional stirring without heating; then washed by decantation and fresh acid added. If no effervescence was perceptible with the fresh acid, it was poured off and the sample washed thouroughly to eliminate the very fine clay particles and any trace of soluble salts. The acid was filtered and used over again several times.

In using filtered acid the residues were carefully watched since fine crystals of gypsum, arising from the combination of the sulphate ions, present as impurities in the acid, with the calcium accumulated from the limestone, in some cases entirely masked the true character of the residue. With a little practice, however, the change of color and con-

sistency of the acid at the point where gypsum started to form could be recognized and the old acid thrown away before the accumulation became detrimental.

The clean residues were washed from the glasses on to pieces of newspaper, dried in an electric oven at a low temperature (60° C.), placed in vials, and labeled. They were studied under a binocular microscope at a magnification of 10× and short descriptions of each residue were written out, giving the percentage of each constituent present. Any microfossils found were removed and placed on numbered slides.

The residue descriptions were plotted on strips of quadrille paper to a scale of 20 feet to the inch and as nearly as possible in their true stratigraphic position, each exposure being on a separate strip. The residues were plotted as bar graphs, with the width of each bar representing thickness of beds sampled and the length of each bar representing the approximate amount of residue present. (Because of the large amount of vein material in the residues, the exact quantity of residue present was not considered very diagnostic of the horizon.) Their character was represented by the coloring of the bars, the proportion of the bar in any one color showing the percentage of the residue formed by a given constituent. At the end of the bar was written the number of the sample and any remarks concerning the peculiar character of that residue which could not be represented by symbols. On the opposite side of the paper strip was written a condensed description of the lithologic character of the beds. A sample diagram is shown as Fig. 3.

Matching of the Sections.—When the attempt was made to match the sections from the different outcrops, no very good accordance could be obtained between them. Then it was discovered that the outcrop descriptions gave the key which enabled one to fit the residue graphs together with some degree of accuracy. First, the attitude of the *Cryptozoa* gave the information as to which was top and bottom (for there are several overturned sections). Then it appeared that all the zones carrying abundant *Cryptozoa* seemed to be at about the same horizon and were very similar. When the sections were arranged with these zones opposite each other, it was found that the residues harmonized very nicely in most places and at a number of horizons showed such close correspondence as to leave little doubt that this was the true correlation of the outcrops. Further work has confirmed this correlation and has shown that most horizons have a general character of residue which can be recognized quite easily if a number of samples are available. However, individual beds may vary considerably from this general character and may resemble beds in other parts of the section very closely. For this reason no correlation can be safely made without a fairly complete residue selection.

GENERAL CHARACTER AND CHIEF CONSTITUENTS OF THE RESIDUES

Shale.—The residues are composed largely of various forms of silica and clay (shale) and have little heavy mineral content. The most abundant constituent is shale, which, in lumps, appears clayey and, when separated, passes through a 100-mesh screen, fulfilling Wentworth's[3] definition of a clay. It varies from a sericitic to a spongy material and, as would be expected, the former is found in greatest abundance in the folded and

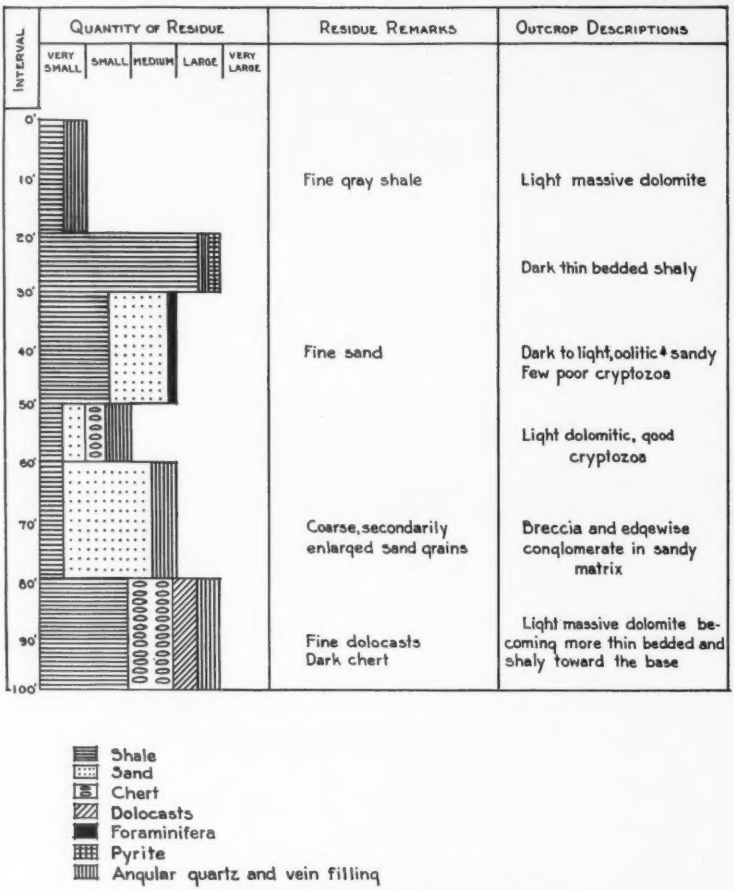

FIG. 3.—Example of plotted residue descriptions.

[3] Wentworth, C. K., Methods of mechanical analysis of sediments: *Iowa Univ., Studies*, vol. 11, no. 11, p. 24, 1926.

most metamorphosed beds. In appearance most of the material is white, flaky, and greasy, but the color may vary from white to almost black. In part, the gray spongy residue may be chert, for it contains very small dolocasts, whose origin will be discussed later. In some cases this shaly material is highly silicified and contains much fine angular quartz and chert.

Sand.—In many of the residues quartz sand occurs, which is a fair guide to some horizons when present in abundance. It is generally rather coarse but varies in size down to material[4] that will pass a 100-mesh screen. Most of the sand grains show secondary enlargement and in some cases they are cemented by silica into quartzite lumps. Some of the grains which happen to be unenlarged are frosted and subangular, indicating a good deal of wear before deposition. A few of the residues contain pink quartz grains, but these are never present in abundance and seem to have no stratigraphic value. Almost all grains are composed of crystalline quartz, and those of chert or milky quartz are extremely rare.

Chert.—The next most abundant constituent of the residues is chert, which is a fair indicator of stratigraphic units when present in abundance. Some of this chert appears glassy, but more often it is cryptocrystalline. It may be either light or dark in color, the latter usually present in larger lumps than the former. Some light chert encloses dark spherules, which have the appearance of replaced oolites but lack the concentric structure. The origin of these spherules is obscure, but it would seem that they are replacements of structures originally contained in the limestones.

While no special study was made to determine the origin of the chert found in the residues, the field and laboratory studies would indicate that the dark chert is largely secondary, as in some cases it encloses patches of limestone with bedding parallel to that outside the nodule. Some of the nodules show slight curvature of the bedding around them, so it seems evident that, while all originated later than the limestone, some must have been formed while the enclosing rock was still soft enough to undergo distortion of the beds during compaction. The origin of the light-colored chert is closely related to the origin of the dolocasts discussed below.

Dolocasts.—Dolocast is a term introduced by McQueen[5] to denote the silicified impressions of dolomite crystals. These impressions show very clearly the rhombohedral outline of the dolomite crystals. They are formed largely of white chert, although some are black to gray, and in some cases they are composed of crystalline quartz. The writer's opinion is that these impressions are probably diagenetic phenomena, and the light-colored chert which forms these impressions took its rise very soon after the deposition of the limestone, and is, for all practical purposes,

[4] Wentworth, C. K., *op. cit.*, p. 24.

[5] McQueen, H. S., *op. cit.*, p. 108.

primary. The dark chert seems to be somewhat later, although no conclusive evidence as to the relative ages of the two kinds of chert has been discovered.

Dolocasts are almost omnipresent in the shaly residues of all these limestones. However, in most cases these dolocasts are very small and can hardly be distinguished under the low power of a binocular microscope. At some horizons large and unmistakable cherty dolocasts appear, which are fairly characteristic of those levels. Unfortunately they are not very persistent, and tend to scatter through a considerable vertical range.

Foraminifera.—The best zonal criteria in these residues are the foraminifera. They are the only recognizable fossils found in most of the limestones, with the exception of *Cryptozoon*, but they have not yet been studied in detail or described. However, certain zones in which they occur have been recognized and form very valuable horizon markers. These foraminifera are all of arenaceous types and are very simple in form. The majority have ovoid tests with simple apertures. Some, however, have tests which are nearly spherical, and many have necked apertures. A very few have as many as three or four chambers and a complex curved neck. The foraminifera occur most abundantly in the lower division of the Allentown, which is correlated with the Upper Cambrian Hoyt limestone of New York State,[6] but some are found at lower horizons. These fossils present a most interesting opportunity for further study in the residues, as they are probably the oldest representatives of their order yet found in this country.

Minor Constituents.—In all the residues there is much angular crystalline quartz, both clear and opaque, which is clearly derived from the filling of small vugs and veinlets in the limestone. It is of little value in stratigraphic studies, for its occurrence depends largely on the amount of shattering that the limestone has undergone. It is useful when it occurs almost alone in a residue, showing the absence of primary insoluble material.

Pyrite, which is rather common in the residues, is also of little value in establishing the stratigraphic sequence. It is found either in its original form or altered to limonite, and almost always occurs as striated cubes or pyritohedrons. It is most abundant in shaly residues and in samples from places where the rock has been shattered, permitting the introduction of iron-bearing solutions. It is clearly secondary and probably was not introduced until late in the orogenic history of the region.

While heavy minerals are scarce and unimportant in these residues, a few grains of various detrital heavy minerals are present. Garnet is perhaps the most abundant, occurring in angular broken fragments. Magnetite and hematite are seen rather frequently, but it is not certain that the hematite is detrital in origin.

[6] Wilmarth, M. Grace, Tentative correlation of the named geologic units of Pennsylvania: *U. S. Geol. Survey*, 1928.

A few grains of zircon have been seen in characteristic square prisms with broken ends. In no bed are the heavy minerals abundant enough to constitute a guide for the identification of the horizons. This scarcity would seem to show that the detrital material in these limestones was derived from a land surface which had undergone long subaerial weathering, destroying all except the resistant siliceous constituents of the igneous rocks.

The stratigraphic column shows the lithology and residues of the various divisions of the series. The chief residue horizons used in mapping are the sandstone near the middle of the Leithsville, the double foraminiferal zone marking the base of the Allentown, and the foraminiferal zone which marks the middle of the Allentown and also marks the top of the zone of greatest *Cryptozoon* abundance. There is no distinct marker at the base of the Beekmantown, either in the field or in the residues, but the change from the sandy residues of the Allentown to the cherty and shaly Beekmantown residues, although gradual, is quite characteristic. The Beekmantown residues often resemble those of the lower Leithsville but usually show many more cherty dolocasts.

SUMMARY AND CONCLUSIONS

1. The Cambro-Ordovician formations of the Lehigh Valley form a series of thin-bedded, unfossiliferous magnesian limestones in which it is difficult to establish stratigraphic markers in the field.

2. The structure of these limestones is so complex that it is very hard to correlate the various outcrops.

3. The insoluble residues used in conjunction with ordinary field methods afford a means of correlating the outcrops and establishing certain definite stratigraphic horizons.

4. Since the residues contain almost no heavy minerals, markers must be established on the basis of the proportions of shale, sand, chert, and dolocasts in the residues.

5. The residues contain the tests of certain arenaceous foraminifera which form good horizon markers at the base and top of the lower division of the Upper Cambrian Allentown formation (Hoyt age), making these foraminifera the oldest yet reported in the country.

In conclusion it may be said that, while the method of insoluble residues can not be recommended for independent use in regions of complex structure, it is very valuable as an adjunct to conventional field methods in determining stratigraphic succession and establishing more exact lithological correlations.

ACKNOWLEDGMENTS

This paper was prepared under the supervision of Dr. F. J. Pettijohn of the University of Chicago along lines originally suggested by Prof. Freeman Ward of Lafayette College. Prof. B. L. Miller of Lehigh University has been more than generous in putting at my disposal material gathered by himself and his students. Mr. M. E. Johnson of the New Jersey Department of Conservation, Geological Division, has also been very kind in communicating to me the results

of an unpublished study of the Cambro-Ordovician limestones of northern New Jersey. Mr. L. E. Workman of the Illinois Geological Survey communicated to me some results of his insoluble residue studies on the Silurian rocks of Illinois and the graphical method of section comparison is due to his work.

Acknowledgment is due Dr. George H. Ashley of the Pennsylvania Geological Survey and to Prof. C. H. Behre of Northwestern University for information concerning exposures in several districts studied. The aid of Prof. R. T. Chamberlin, Dr. C. M. Langton, and Mr. H. A. Ireland in reading and criticizing the maps and manuscript is gratefully acknowledged.

JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 5, NO. 3, PP. 133–136, FIG. 1, DECEMBER, 1935

AN "ANTIDUNE" PHASE OF EOLIAN RIPPLE MARKS[1]

BRADFORD WILLARD
Pennsylvania Topographic and Geologic Survey, Harrisburg, Pennsylvania

ABSTRACT

The "antidune" phase of ripple marks produced in water currents is a well-known phenomenon. It has been reported also as produced by high winds moving dry snow at low temperatures. An "antidune" phase recently observed in wind-blown sand is here described.

Wind-formed or eolian ripple marks are a common feature of sands most often seen on beaches above high water mark and in dunes. These ripple marks are usually characterized by low amplitude (height) in relation to length as contrasted with water-formed ripple marks of which the amplitude is commonly greater proportionally. Thus, the eolian type has a higher ripple index. The normal or commonest eolian and water-current asymmetric types resemble one another in that the steep face is to the leeward in the former and down stream in the latter. This normal type of ripple mark is produced under conditions of moderate currents. With an increase in velocity, the ridges tend to disappear, and instead there is produced a smooth surface of drifting sand, the so-called "smooth phase."

With further increase in velocity of water currents beyond that productive of the smooth phase, reverse or "antidune"[2] ripples are produced. These have the steep slopes up stream; the gentle slopes down stream. Apparently, an analogous "antidune" phase has not been generally observed in the case of a high wind moving *sand*, although such a condition has been recorded for rapidly driven *snow*. This phenomenon is described by Kindle and Bucher[3] as follows:

> WIND RIPPLE MARK ON SNOW. In cold climates wind ripple mark is sometimes formed on dry and granular snow. The cohesion of snow flakes or particles varies with the temperature and consequent moisture, so that ripple mark forms on snow only at temperatures considerably below the freezing point. With low temperature and the right type of powdery snow, a winter gale almost always develops over parts of a large level expanse of snow a type of asymmetric ripple with a highly variable wave length, which differs conspicuously from eolian sand ripple marks in having its steep face on the windward side instead of the leeward side. Symmetrical ripple mark with a wave length of

[1] Published with the permission of the State Geologist of Pennsylvania.

[2] Gilbert, G. K., The transportation of débris by running water: *U. S. Geol. Survey, Prof. Paper* 86, p. 31, 1914.

[3] Kindle, E. M., and Bucher, W. H., Ripple mark and its interpretation: in Twenhofel's *Treatise on Sedimentation*, 1932 edition, 643.

several inches sometimes forms on snow, but it is a relatively rare phenomenon. Obviously the shape of the ripples as reported in these observations is due to peculiar conditions inherent in the properties of snow which have not been analyzed so far.

trends nearly east-west,[4] and is of the type commonly found along the New Jersey coast. A gently seaward-sloping expanse of clean, quartz sand extends from the water's edge up to

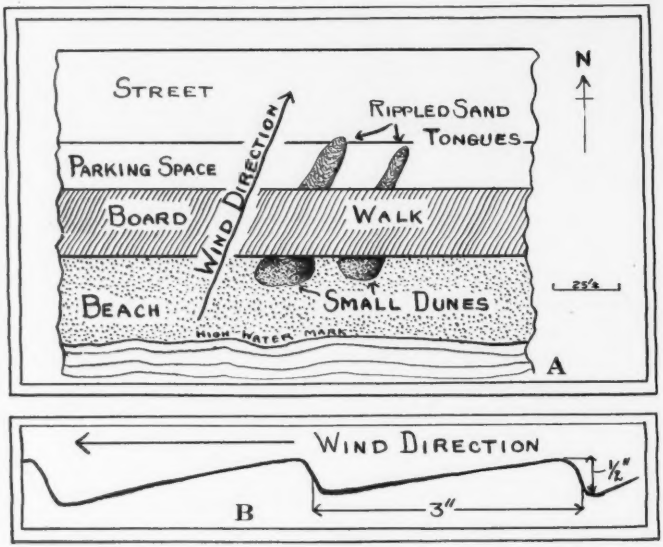

FIG. 1.—A, Sketch map of a part of beach showing position of ripple-marked sand tongues. B, Cross-section of ripples.

Be this as it may, it appears that the "peculiar conditions inherent in the properties of snow" are not the only factors responsible for the production of an eolian "antidune" phase since this type has been seen recently by the writer in certain wind-formed ripple marks in sand. During September, 1934, the author was on vacation at Cape May, New Jersey. On September 4 a strong south-southwest wind, reaching at times almost gale velocity, blew for several hours. Cape May beach the ubiquitous board walk. This structure is about 25 feet wide and is supported on posts 3 or 4 feet high. It divides the beach proper from an automobile parking space and a paved street respectively parallel to the walk northward. The wind blowing inshore and almost at right angles to the beach tended to move fine sand inland. Apparently the smooth phase of transportation following

[4] The reader is referred for the local geography to *New Jersey Dept. Conservation and Development*, Atlas Sheet (topographic) no. 37.

that in which normal ("dune" phase[8]) ripples are produced obtained on the beach between high water mark and the board walk (see Fig. 1), for that area was quite smooth. However, at certain points along the board walk concentration of air currents over limited areas materially increased the wind velocity locally. It was at such points that an "antidune" phase was observed.

Figure 1A shows roughly a plan of a section of the beach and adjoining property where this phenomenon was seen. Along the upper edge of the beach adjacent to the board walk are occasional, small dunes, some of them topped by coarse beach grass, but nevertheless capable of being eroded by wind, though obviously above the reach of waves save perhaps those of exceptionally violent storms. The strong wind sweeping past and among these on the date mentioned caught up the fine dune sand and possibly some of the smaller particles from the beach also, and carried it through the low, confined space beneath the board walk whence it was deposited as long, low tongues extending out from under the opposite (north) side of the walk across the parking space and even onto the street pavement in some examples. Usually a tongue was produced opposite each dune, but smaller ones developed at other points for no very obvious reason except perhaps the presence of a supply of fine sand. Evidently the wind confined in the small space beneath the walk concentrated its energy upon the movement of relatively fine sand, derived chiefly from the dunes, but this increased transporting power was rapidly lost upon emerging from the leeward (landward) side of the walk with attendant loss of burden. It is noteworthy in regard to this last statement to observe that the tips of the tongues tended to lose their ripples and assume the smooth phase as might be expected with diminution of wind velocity. (This is not shown in Fig. 1 as the smooth area was very small.)

The sand of the tongues was very fine, almost powdery, much finer than the ordinary beach sand, since it was derived chiefly from the dunes. Microscopic examination of it was impossible under the circumstances. Each tongue of sand was seen to be ridged by a succession of ripple marks with convex sides to windward, that is, toward the beach. Furthermore, the windward side of each concentric or chevron-shaped ridge was the steeper, the leeward slope being quite gentle (Fig. 1B). The ripples of this type clearly belonged to the "antidune" phase. They had a length of approximately three inches and an amplitude of about one-half inch. This contrasted strikingly with wind ripples of the normal or "dune" type measured on the same beach a few days previously. In these the length was nearly the same, but the amplitude only about one-sixteenth of an inch. Movement of the "antidune" ripples was not observed, but was assumed to be to windward.

From the foregoing observations it appears that the "antidune" phase

[8] Gilbert, *idem.*, p. 31.

of ripple marks not uncommonly observed in water-borne sands and known also to be produced by high winds moving dry, powdery snow at low temperatures, may also occur in wind-worked sandy deposits under exceptional conditions where fine sand and strong wind are involved. However, the phenomenon seems to be comparatively rare, and in this instance was observed to take place very locally under quite specialized conditions which had brought about a concentration of transporting power of the wind into a small space where sand of the requisite fineness and dryness was plentiful.

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A TEST FOR THE SIGNIFICANCE OF LITHOLOGICAL VARIATIONS

CHURCHILL EISENHART
Princeton University, Princeton, New Jersey

ABSTRACT

It is pointed out that the notion of "homogeneity" is dependent on the classification used. Sampling fluctuations are such that two or more samples are rarely identical for any classification, and, therefore, some criterion is needed to indicate plausibility of homogeneity in a given instance. The Chi-Square test is an appropriate criterion, and it is easily applied to geological data, as is shown in some worked samples.

Statement of the Problem.—Geologists believe "that a sediment must carry with it definite clues of its past history." If this be assumed as a working hypothesis, what are its consequences, and how may its tenability be tested?

One consequence of this hypothesis is that, if it be true, then two or more samples of sediments which are *known* to have had the same past history (i.e., are known to have been drawn from the same parent deposit) ought to show certain similarities. That is, they ought to be "homogeneous" with regard to certain attributes.

In the second place, if two or more samples have been taken from parent deposits which are *known* to be different, then the samples ought to show significant differences with regard to some attribute(s). Moreover, a test designed to indicate whether or not samples are similar with respect to these attributes (i.e., can be considered as coming from the same parent population) should, in the present case, assert dissimilarity on the basis of the information contained in the samples.

If homogeneity is plausible, one can proceed to estimate the true means, medians, measures of dispersion, etc., of the parent deposit in order to make comparisons with other deposits. But one can estimate none of these unless the data are *comparable*, and this implies that group A is composed of homogeneous items, and that group B is also homogeneous—a group composed of dogs and chickens cannot be compared with another composed of cats and ducks and result in a meaningful answer. The notion of homogeneity is fundamental.

Homogeneity.—According to the dictionary a group is *homogeneous* if it consists of parts all of the same kind. Obviously, things that are identical are also homogeneous; but identity is not necessary for homogeneity. To take an example, the group "native white, age 35, of New York City" is homogeneous in respect of color,

nationality, age, and place of residence, but the individuals are not all identical as some may be of native and others of foreign parentage. Identity implies homogeneity in all respects, and hence is rarely, if ever, realized in practice. Moreover, homogeneity depends on the classification used—the group above is not, off hand, homogeneous in respect of parentage.

Testing for Homogeneity.—Since homogeneity is dependent on the attribute(s) under consideration, it is of interest to know how to test for homogeneity in a given case.

The simplest case is that of a single sample. In respect to a chosen attribute the sample either *is* homogeneous with regard to the attribute, or *it is not*—there is no half way point. Suppose the lithology of a sample of pebbles is of interest. If they are all limestone pebbles, the sample is homogeneous; if they are mostly limestone, but also some shale pebbles are present, then it is non-homogeneous; it is, however, homogeneous if we are using the coarser classification "igneous—not igneous."

Proceeding to the case of two samples, if both samples contain limestone and shale pebbles and no others, under what conditions can the two samples, considered together, be regarded as lithologically homogeneous? What about the proportions? If they are not identical samples, some criterion must be used to assist one in deciding whether or not the two samples are homogeneous in the sense that they have both come from the same parent deposit, whose mineral proportions are approximated by the proportions in the samples, the variation being due to sampling fluctuations.[1]

In the absence of knowledge other than that contained in the samples

[1] The need for some mathematical criterion in problems of this sort has been appreciated by Lincoln Dryden and in a recent paper (A statistical method for the comparison of heavy mineral suites; *Am. Jour. Sci.*, (5), Vol. 29, No. 173, pp. 393–408, May 1935) he has proposed using the correlation coefficient, r, as a measure of the similarity of the various *proportions* in the samples to be compared. Unfortunately he was unaware of the pitfalls of the method he selected.

As will become more evident further along in this paper, *it is necessary* to take into account the *sizes* (number of items in the respective samples) of the samples under consideration when testing for similarity. As the size of a sample is increased the proportions of the various minerals in the sample tend to approximate the proportions of the parent deposit with greater and greater stability, that is, sampling fluctuations will account for less and less of observed discrepancies in proportions as the sample size is increased. Dryden's method does not take this into account, as he uses the percentages of the various types of minerals in his samples and makes no mention of the total sizes. A short reference to samples A and B from page 395 of Dryden's paper will emphasize the necessity of utilizing sample sizes: the two samples do not differ greatly as regards the percentages of their mineral contents, and, if these samples are assumed to contain about 300 items each, the probability is .4 that the observed differences are ascribable to sampling fluctuations, but, if it is assumed that the samples contain about 3000 items each, the corresponding probability is .00000 00000 002213 which implies that it is practically impossible to ascribe the dissimilarities to chance.

Furthermore, *the coefficient of correlation, r, is not applicable* to samples classified in respect to mineral composition. This coefficient can in general, be used only in connection with attributes which are measurable or quantitative; it can be used with qualitative characteristics only when these can be ordered in a numerical sense (e.g. poor, fair, good excellent). As mineral classification is purely descriptive and unordered, the correlation coefficient is unserviceable, and it is necessary to turn to the theory of probability in order to obtain a criterion to test homogeneity in respect of lithological composition.

themselves, the question of whether or not they are homogeneous in a statistical sense depends on whether or not their differences can be ascribed wholly to chance fluctuations. In drawing inferences from data one must always accept a certain risk of being wrong, the amount of risk acceptable depending on the purpose for which the sample information is to be used and on the rashness of the observer. For example, what risk is assumed if the two samples hereinafter are considered as homogeneous in a statistical sense?

Sample	Limestone	Shale
α	103	794
β	109	781

The table gives the frequencies of these two types of pebbles as found in samples α and β. The size distinction which determines whether or not a particle is a pebble being the same in each case—i.e. the data are comparable.

An appropriate test (the Chi-Square Test, to be discussed hereinafter) asserts that in drawing many pairs of samples at random from the same parent deposit, one would find between the two samples such variations as were observed or worse in approximately sixty-two percent of the cases. In other words, in a single trial such variation or greater variation is very likely—from a sampling point of view it has a probability of 0.62. Hence one does not run any great risk in assuming that α and β are samples drawn from the same parent deposit.

On the other hand, if samples α_1 and β_1 are being examined, samples whose frequencies are

	Limestone	Shale
α_1	180	520
β_1	96	655

the same test asserts that about once in 10^{15} samples would one find such (or a worse) irregularity between samples drawn from the same parent population. In such a case the hypothesis that they are homogeneous is untenable, and should be rejected.

This criterion (the Chi-Square Test) is very generally applicable, and may be applied to a group of several samples with many categories in each sample (the same categories, of course). It has been used, and is being used, with great success in anthropometric, biometric, and econometric studies. Therefore it seems fitting to discuss it in this paper with the view of adopting it as a criterion in certain geological problems involving sampling.

The Chi-Square Test.—For simplicity, let it be assumed that one has only two samples, S_1 and S_2, and that one has classified some attribute into two well-defined categories C_1 and C_2. A table can now be formed

Sample	Attribute		Totals
	C_1	C_2	
S_1	(S_1C_1)	(S_1C_2)	(S_1)
S_2	(S_2C_1)	(S_2C_2)	(S_2)
Totals	(C_1)	(C_2)	N

where ( ) denotes summing: thus (S_1C_1) denotes the total number of items from sample S_1 which possess the attribute C_1; likewise (S_1) denotes the total number of items in sample S_1; N is the total number of items in the sample, $N=(S_1)+(S_2)=(C_1)+(C_2)$.

If one now assumes that both samples are from the same parent universe, one expects to find the same proportion of, say, C_1 in sample S_2 as in sample S_1 (i.e., the proportion should be independent of the sample chosen). In the present notation this is expressed by

$$\frac{(S_1C_1)}{(S_1)}=\frac{(S_2C_1)}{(S_2)}. \qquad (1)$$

Moreover, since $(S_2C_1)=(C_1)-(S_1C_1)$ and $(S_2)=N-(S_1)$, substitution in (1) yields $[N-(S_1)](S_1C_1)=(S_1)(C_1)-(S_1)(S_1C_1)$, which reduces to

$$(S_1C_1)=\frac{(S_1)(C_1)}{N}, \qquad (2)$$

a relation which enables one to calculate the frequency of a cell on the assumption of complete independence. Since the total of the first row is to be (S_1), evidently $(S_1C_2)=\dfrac{(S_1)(C_2)}{N}$ giving $(S_1)=\dfrac{(S_1)[(C_1)+(C_2)]}{N}$. Consequently, if the proportions are to be truly independent the various frequencies must be distributed as in the following table:

Sample	Attribute		Totals
	C_1	C_2	
S_1	$\dfrac{(S_1)(C_1)}{N}$	$\dfrac{(S_1)(C_2)}{N}$	(S_1)
S_2	$\dfrac{(S_2)(C_1)}{N}$	$\dfrac{(S_2)(C_2)}{N}$	(S_2)
Totals	(C_1)	(C_2)	N

The reasoning is perfectly general, and if one has samples $S_1, S_2, \cdots, S_i, \cdots$ and categories $C_1, C_2, \cdots, C_j, \cdots$ then the frequency appropriate to complete independence for the cell in the jth column of the ith row is

$$(S_iC_j)=\frac{(S_i)(C_j)}{N},$$

the Independence Frequencies. (3)

Denoting the *Observed Frequency* by $(S_iC_j)'$, due to sampling fluctuations $(S_iC_j)'\neq(S_iC_j)$, and the difference

$d_{ij}=(S_iC_j)'-(S_iC_j)$ can be formed. (4)

It is evident that some d's will be plus and others minus, and that the sum of all the d's will be zero since

$$\sum_{ij}(S_iC_j)'=\sum_{ij}(S_iC_j)=N,$$

and hence any criterion must depend on the values of the d's without regard to sign.

A mathematical analysis of the problem indicates that an appropriate criterion is the Chi-Square Test, based on

$$\chi^2=\sum_{ij}\frac{(d_{ij})^2}{(S_iC_j)} \qquad (5)$$

in which d_{ij} is defined above in (4), leading to a probability measure of the homogeneity of the data. It is evident that the nearer the values observed are to the independent frequencies, the smaller the χ^2. Consequently the magnitude of χ^2 is an indication of the degree of departure from perfect homogeneity.

It has been shown by R. A. Fisher (Phil. Tran., Vol. 222A, p. 357; Jour. Roy. Stat. Soc., Vol. XXXVII, p. 447) and by others that for *large samples* the probability distribution of χ^2 defined by (5) is approximately the same as that of another χ^2 defined years before by Karl Pearson in con-

nection with Goodness of Fit. Tables are available (Fisher's *Statistical Methods for Research Workers*, and Pearson's *Tables for Biometricians and Statisticians*) which give the probability of obtaining by chance a value of χ^2 as great or greater than that actually observed in sampling from truly homogeneous material. In using these tables of probability one looks in the table under the number of degrees of freedom, n, given by

$$n=(r-1)(c-1) \qquad (6)$$

for a table of data consisting of r rows and c columns.[2] As Pearson's table was originally constructed for another purpose, one must enter his table with $n'=n+1=(r-1)(c-1)+1$; the present notation is the same as Fisher's. For a fuller discussion, including short cut methods applicable to special cases see Chapter IV of Fisher's book.

Finally, it must be remembered that the tables of probability have been calculated on the assumption of large samples. It has been found that this calculated distribution is not very closely realized for very small class frequencies, and, therefore, to play safe one should try to have the smallest independence frequency greater than 10 (a minimum). Nothing is gained by including nearly vacuous classes; it is far better to throw several classes together and enter the probability table with a correspondingly smaller number of degrees of freedom.

[2] It was seen above that in a 2×2 table, when one value has been calculated, all the rest are determined, since the marginal totals are to remain the same. In general, as can readily be seen by trial, in a table of r rows and c columns, there are only $(r-1)(c-1)$ frequencies that need be calculated, the rest then being obtained by subtraction from the marginal totals.

Worked Examples (with comments).—(1) The two samples α and β referred to above will afford data to illustrate the application of the Chi-Square technique. In the tabulation immediately following the upper number in a cell is the observed frequency, and the number in ( ) the corresponding independence frequency.

Sample	Attribute		Totals
	Limestone	Shale	
α	103	794	897
	(106.42)	(790.58)	
β	109	781	890
	(105.58)	(784.42)	
Totals	212	1575	1787

In obtaining the independence frequencies only one value had to be calculated, e.g., $\frac{212\times 897}{1787}=106.42$, the others being obtained at once by subtraction from the marginal totals. Consequently, there is only one degree of freedom, $n=1$.

In the table which follows, the upper number is the absolute value of the difference between the observed and the expected frequency; the lower number is the square of the difference just mentioned divided by the expected frequency $\left(\text{e.g.,}\frac{(103-106.42)^2}{106.42}=\frac{(3.42)^2}{106.42}=.109^+\right)$, and this is the contribution to χ^2 from this cell of the table. By adding

the separate contributions it is found that $\chi^2 = .249$. By interpolating between values in Fisher's table, for

Sample	Li	Sh	Totals
α	3.42 .109	3.42 .015	.124
β	3.42 .110	3.42 .015	.125
Totals	.119	.030	$.249 = \chi^2$

$n = 1$, one finds a corresponding probability of .62 which means that sixty-two times in a hundred one ought to obtain an equivalent or larger value of χ^2 in drawing two such samples from a common parent population. One assumes no great risk, therefore, in accepting α and β as samples from the same parent deposit and in asserting that they are homogeneous in that sense.

(2) Proceeding in the same way with samples α_1 and β_1 the contributions of the four cells to χ^2 are found to be:

Sample	Li	Sh	Totals
α_1	25.84	5.53	31.37
β_1	20.93	5.33	26.26
Totals	46.77	10.86	$57.63 = \chi^2$

These individual contributions are so great that it is not in the least surprising to find that the probability of such a χ^2 (or a larger one) is of the order 10^{-15}. In such a case one rejects at once the hypothesis of homogeneity, the probability by chance of such a variation from homogeneity of occurrence being so small.

It may be asked: "Where is the border-line beyond which a hypothesis should be rejected?" There is no absolute answer to this question, but, in general, experience has shown that if P is less than .05 it is wise to reject the hypothesis. Of course, in doing this, one will, once in twenty times, consider as non-homogeneous two samples that really are homogeneous. If extremely desirous not to lose these samples, $P = .01$ can be chosen as the criterion point, but it must be remembered that in so doing one admits to the homogeneous class samples that are really non-homogeneous and which would have been excluded by using $P = .05$. Every experimenter must decide for himself what he will accept.

What if $P = .99$? In this case the hypothesis ought to be rejected, in general, because the probability of choosing samples that agree so well from all possible samples is so small that one suspects that they were not *random*. If, however, it is certain that no bias has entered, then homogeneity can be accepted, but with a grain of salt, remembering that such luck happens only once in a lifetime.

(3). As a further illustration, interesting results follow the application of the χ^2 method to a problem treated by Dryden (loc. cit. p. 402 ff.). While examining a contact between Eocene and Miocene beds in Maryland, Dryden selected samples from about one foot below the contact (in the Eocene) and about a foot above the contact (in the Miocene). Off hand the figures seemed to indicate that some of the Eocene material had

been reworked into the base of the Miocene bed, the Eocene and Miocene samples showing certain strong similarities. An application of the χ^2 method to Dryden's first data indicates at once that the Eocene and the Miocene beds are distinctly different in their mineral make up, the value of χ^2 obtained being nearly three times the one per cent value.

Apparently unfamiliar with the χ^2 criterion, and not liking the hypothesis regarding the reworking of Eocene material into the Miocene, Dryden "decided to find in a little more detail just where and how the mineral change from typical Eocene to typical Miocene took place. With a rather hazy idea of what was demanded in the way of samples, collections were made from the top inch of the Eocene (if there had been reworking at this place the material farther down could have had nothing to do with it), in one-inch intervals from the contact to six inches above the contact, and one sample from 6–9 inches above and one from 9–12 inches above the contact." He gives the percentages of various minerals by count in his Table V. These are reproduced below using 4 categories in place of his 9 on account of the paucity of data in certain categories. The corresponding frequencies (rounded to integers) are given on the assumption of samples in the neighborhood of 300 items (Dryden has informed the writer that this is the general size of his samples). Calculating χ^2 for the whole table one finds $\chi^2 = 96.30$, of which 55.93 is contributed by the Eocene sample. For $n = (9-1)(4-1) = 24$, the one per cent value is 42.9, and since the observed value is far greater than this, one concludes that the Eocene and Miocene samples are not collectively homogeneous in respect to heavy minerals. Since more than half of the total χ^2 comes from the Eocene sample, it is of interest to examine the situation with this sample omitted.

Calculating χ^2 for the complete table less the Eocene sample, one finds $\chi^2 = 32.54$ with $n = (8-1)(4-1) = 21$. From tables it is found that $P(\chi^2) = .05$, and in view of the fact that the data has been rendered more homogeneous by bunching categories together (for by adding together pairs like 5, 9 and 9, 5 one gets 14,14 which is more homogeneous) the evidence suggests that *all* the samples of Miocene material cannot collectively be considered as homogeneous in respect to heavy

	Eocene		Miocene													
	−1,0		0,1		1,2		2,3		3,4		4,5		5,6		6,9		9,12	
	%	*f*	%	*f*	%	*f*	%	*f*	%	*f*	%	*f*	%	*f*	%	*f*	%	*f*
Z	50	150	28	84	30	90	30	90	28	84	31	93	26	78	29	87	41	123
St	24	72	36	108	33	99	31	93	33	99	33	99	32	96	29	87	25	75
G	6	18	20	60	18	54	20	60	20	60	18	54	22	66	17	51	19	57
All others	20	60	16	48	19	57	19	57	19	57	18	54	20	60	25	75	15	45

All percentages add to 100, all frequencies to 300.

minerals. In addition, an examination of the individual contributions shows that 17.57 (more than half) of the total χ^2 came from the [9,12] sample. If this sample were removed it is clear that the remainder of the Miocene samples could be regarded as homogeneous.

A further investigation of the [9,12] sample yields:

A Table of Comparisons of the [9, 12] Sample with

Sample		χ^2	n	$P(\chi^2)$
(Eocene)	−1,0	25.16	3	much less than .01*
(Miocene)	0,1	13.47	3	less than .01
(Miocene)	6,9	14.89	3	less than .01

(As a comparison, Miocene [0,1] and [1,2] when compared yield $\chi^2=1.68$, giving $P(\chi^2)=.65$.)

* For $n=3$, $P=.01$ when $\chi^2=11.34$.

These figures suggest that sample [9,12] is

(a) significantly different from the Eocene sample,

(b) significantly different from the Miocene samples,

and

(c) that (b) is not due to a "distance effect."

Considering in connection with this the other evidence of Dryden it appears that there is a sudden change in heavy mineral content in the neighborhood of 9 to 12 inches above the Eocene-Miocene contact. Does this change continue farther up, or does the composition revert to that in accord with samples [0,1] to [6,9]?

Before summing up, there are several warnings that need to be explicitly stated: First, the χ^2-test should not take the place of common sense; simply because two samples from widely separated regions yield a small value of χ^2 it should not be concluded at once that they have had a common geologic history. Secondly, the χ^2-test is not omnipotent; samples may be homogeneous, say, in respect of lithology (as determined by the χ^2-test) and decidedly different in respect of shapes of particles. In such a case it is up to the geologist to decide whether mineral content or particle shape is more important—after all complete similarity can rarely be established. Finally, the χ^2-test *cannot be applied to data where the units are weights, lengths, etc., as it is only applicable to frequencies.* If applied to weights, for example, it would correspond to saying that a given unit of weight, say a gram, at all times retained its identity and never dispersed—i.e., if a gram of material were colored pink, put back in the main pile, the material mixed, then one should expect to find all the pink particles still all together forming one unique indispersible unit. Of course, this difficulty does not arise in the case of pebbles counts, as one pebble at all times retains its identity and cannot fall apart into smaller particles.

SUMMARY

The meaning of "homogeneous" is arbitrary, and in a given case needs to be defined with care, since it will depend on the classification used. An appropriate criterion to test for homogeneity corresponding to a certain classification is the Chi-Square Test. The test can be used to indicate whether a given deposit is homogeneous throughout in a statistical sense,

and it will also indicate when applied to several deposits whether they can be considered as mutually homogeneous. Since homogeneity is intimately connected with comparability of data these concepts must be fully understood if conclusions due to non-comparable data are to be avoided.

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NOTES

DISCOVERY OF BENTONITE IN EUROPE

W. H. TWENHOFEL

Some years ago the writer predicted that bentonite would be discovered in Europe as soon as European geologists became familiar with the characteristics of the rock and made search therefor. This prediction has now materialized. The discovery was made and the occurrence is described by the Polish geologist Zbigniew Sujkowski (The Bentonite in Poland, with an English summary, Archive de Minéralogie de la Société des Sciences et des Lettres de Varsovie, vol. 10, 1935, pp. 98–116).

The bentonite was discovered in the lower Sarmatian (Miocene) of Volhynia in Poland. There is a single bed ranging from 5 to 20 cm. in thickness. The enclosing rocks are sandstones and the clay mineral in the bentonite is stated to be montmorillonite. The rock is described and its distribution given. The occurrence is interpreted as representing a fall of volcanic ash in the late Miocene, the ash supposedly having been derived from volcanoes connected with the vulcanism of the Siebenburgen district of Germany.

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THE USE OF ACETIC ACID TO OBTAIN INSOLUBLE RESIDUES

DONALD W. ST. CLAIR
Syracuse University, Syracuse, New York

ABSTRACT

Insoluble residues are valuable for correlation in that they contain detrital and authigenic minerals and structures such as dolocasts. Acetic acid is shown to give a more satisfactory residue from limestone than hydrochloric acid, in that it preserves a more complete mineral suite; leaves a support for the frail silica cast; and provides a clean residue, without forming a gel, flocculated clay or organic coating. Some results are given of work with Upper Silurian limestones from Syracuse, New York.

PURPOSE OF INSOLUBLE RESIDUES

The establishing of characteristic mineral suites of limestones to bring about a physical correlation has generally involved a hydrochloric acid digestion with several ends in view: (1) To obtain a representative insoluble residue; (2) to concentrate detrital and authigenic mineral grains, and (3) to preserve secondary structures, as dolocasts, which may be characteristic for certain horizons.

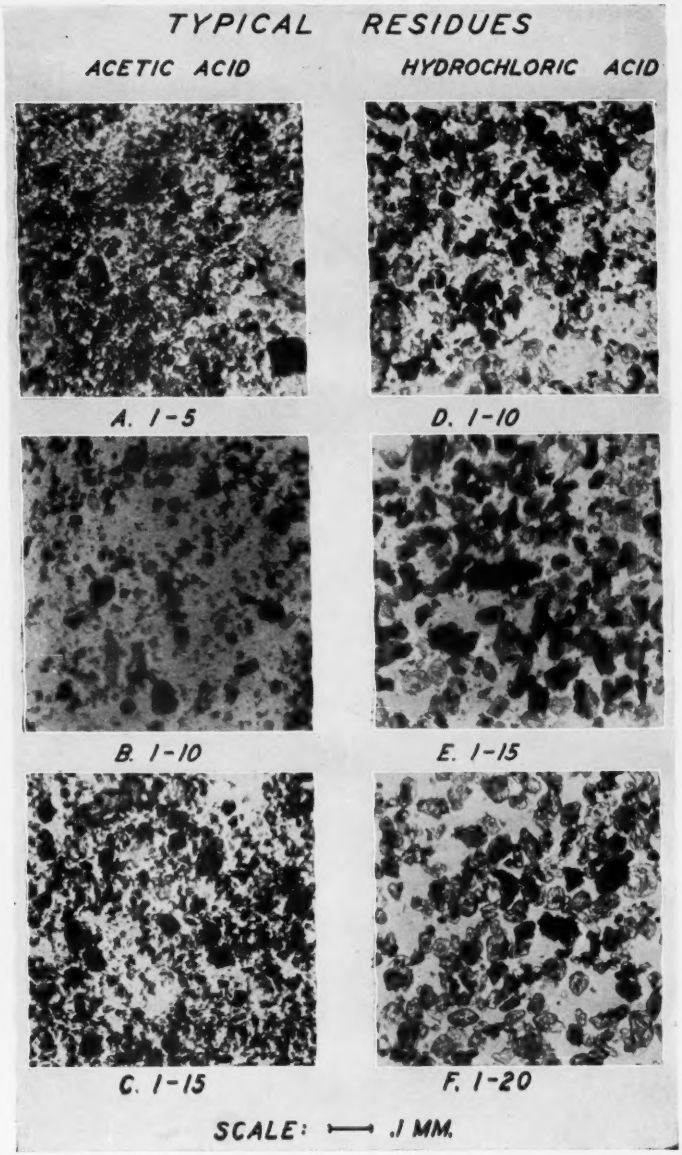
The necessity of concentrating the few diagnostic grains from a large sample is obvious, and since little of correlative value has as yet been gained from the mineralogical study of the preponderant calcium carbonate, the insoluble residue method alone remains to give results.

Many minerals other than carbonates, as apatite, certain feldspars, pyrrhotite etc., are soluble in even weak solutions of an acid as powerful as hydrochloric, and hence do not appear in the insoluble residues.[1] Thus, they neither contribute to the story of sediment provenance nor add to our knowledge of authigenic mineral suites. Wholesale destruction of dolomite crystals ignores a potentially valuable criterion of limestone correlation, in addition to removing the support from the frail siliceous coating to which the name

[1] For a complete discussion of the solubility of minerals in acids, the reader is referred to Dr. F. Steinreide's *Anleitung zur Mineralogischen Bodenanalyze*, Leipzig, 1921, and the well known works on sedimentary petrology.

EXPLANATION OF PLATE 3

A—Acetic. Dark dolomite rhombs prominent, with fine unflocculated clay making up groundmass.
B—Acetic. Dolomite rhombs and some large opaque grains. A little fine clay is present.
C—Acetic. Large pieces are nearly all dolomite, finer material is unflocculated clay.
D—Hydrochloric. Opaque masses of clay, transparent grains are mostly quartz.
E—Hydrochloric. Characteristic lumping of clay.
F—Hydrochloric. Stable grains persistent, obscured by aggregates of clayey particles.


TYPICAL RESIDUES
ACETIC ACID
HYDROCHLORIC ACID
A. 1-5
D. 1-10
B. 1-10
E. 1-15
C. 1-15
F. 1-20
SCALE: .1 MM.

dolocast has been applied.[2] As a result of this removal the silica casts break up easily and are not recognized in the residue.

ACETIC ACID AS A SOLVENT

Acetic acid has been used as a less destructive solvent, one which will accomplish the needed concentration of mineral fragments, preserve the mineral complex more completely, facilitate the examination of siliceous structures, and lead to cleaner residues and more accurate results.

Samples of the Jamesville formation, Upper Silurian, Helderberg (?), from East Onondaga, two miles south of Syracuse, New York, were used in this study. The Jamesville is a massive, practically unfossiliferous limestone, a sample from the same horizon as that used in this study analyzing 3.47 per cent $MgCO_3$ and 92.79 per cent $CaCO_3$.[3]

Several dilutions of acetic and hydrochloric acids were experimented with. Digestions were made with solutions having the concentrations listed below:

Glacial Acetic Acid (99.8 per cent)
1–5, 1–10, 1–15 parts acid to water.

Commercial Hydrochloric Acid
(29.4 per cent)
1–10, 1–15, 1–20 parts acid to water.

The following points in favor of the acetic acid treatment were noted during the digestive process.

(1) The production of a sticky, insoluble carbonaceous film is common from the hydrochloric acid digestion of this limestone. Acetic acid does not flocculate these probably organic particles, so no troublesome coating of mineral grains ensues. Time saved in the direct production of clean samples will offset the somewhat longer time needed for digestion in the weaker acid.

(2) With this limestone prolonged action of even dilute hydrochloric acid forms a gelatinous material which is filtered off with the insoluble portion. Aside from the inaccuracies inherent in such treatment, dolocasts may be attacked, and the presence of the gel impedes deflocculation of the residues.

(3) The same lack of flocculation is noted with clay particles. Partly due to the gel mentioned above, these particles are often cemented on and aggregated with detrital minerals. Even after repeated washing and attempted chemical deflocculation, grains from an hydrochloric acid digestion remain coated with clay and silt, making heavy residues particularly difficult to study.

ILLUSTRATIONS

Photomicrographs of residues from each of the six acid dilutions are shown. In the acetic acid residues the perfect preservation of dolomite rhombs can be seen together with other minerals of large size, and a fine groundmass of individual clay particles. In the hydrochloric residues the stable grains stand out, but the complete mineral suite is missing. Also the clay has flocculated and obscured many small grains.

[2] McQueen, H. S., Insoluble Residues as a Guide in Stratigraphic Studies, *Missouri Bureau Geol. and Mines*, pp. 102–31, 1931.

[3] Analysis for the author by N. W. Thibault, Virginia Polytechnic Institute, Blacksburg, Va.

DOLOCASTS

The treatment for recovery of dolocasts is quite simple. The residue is divided into light and heavy portions with commercial bromoform (S.G.-2.8). The heavy separate is split again with methylene iodide (S.G.-3.3) which yields a light portion mostly of dolomite (S.G.-2.8). The specific gravity of the dolomite will vary with the amount of silica present in the dolomite-dolocast composite. If the operations are carried out by centrifuge, the dolomite separation will be quite definite.

The dolomite portion can be dissolved on a watch glass with dilute hydrochloric acid and the solution controlled by adding water or acid. The silica casts are obtained in an unbroken condition, and with care, very fragile structures may be recovered.

RESULTS

These tests were conducted to find the acid concentration which gave the best balance between cost, time and harshness of chemical action. A 1–7 dilution (about 2N) of acetic acid, which may be strengthened to shorten the time of digestion, is suggested.

The possible additions to known mineral suites are many, the present paper suggests a method of procedure for obtaining them.

CONCLUSIONS

Acetic acid is superior in some respects to hydrochloric acid for obtaining insoluble residues from limestones, because:

(1) It preserves the mineral suites more completely.

(2) It facilitates the examination of dolocasts by not attacking the dolomite crystals which support the silica cast.

(3) Although digestion may take somewhat longer, the residue is cleaner since (a) no gelatinous material is formed (b) carbonaceous or clay particles are not flocculated.

Thanks are due Dr. Earl T. Apfel of Syracuse University for suggesting the line of investigation which led to these results.

BOOK REVIEWS

FILIPESCO, M. G., *Sur la nature des roches siliceuses de L'Éperon de Vălenii de Munte*, Bull. de la Section Scien., Acad. Roumaine, XIII-ème Année, No. 6, 1930 (7 pages).

FILIPESCU, M. G., Note sur la présence de roches siliceuses d'origine organique dans les Couches de Podul Morii, à Vălenii-de-Munte, distr. de Prahova, C.R. des séances de l'Institut Géologique de Roumaine, XIX, 1931 (5 pages).

FILIPESCU, M. G., Sur les roches siliceuses d'origine organique et chimique de l'Oligocène des Carpathes roumaines, C.R.t. 192, 1931 (3 pages).

FILIPESCO, M. G., Sur les accidents siliceux des couches de Tisaru (Flysch des Carpathes roumaines), C.R.t. 194, 1932 (3 pages).

FILIPESCO, M. G., Quelques phénomènes de diagenèse dans les sédiments du Flysch compris entre Teleajan et Doftana, Buletinul Labor. de Miner. al Univ. din Bucuresti, vol. I, 1934 (13 pages).

The conclusions arrived at by Filipescu (or Filipesco) will serve to show the theme which runs through the papers named above: (1) Siliceous beds (of the areas named) of a cherty, opaline, or menilitic character are found only at horizons which are rich in diatoms, sponge spicules, radiolaria, or in some combination of these three; (2) the silica comes from these organisms, and may be found either as skeletal remains, or may be dissolved and redeposited as lenses, replacing other material (especially calcareous cement), or in other forms; (3) diatoms contain oil, and this oil may be transformed into ordinary petroleum (refers to Tolman and others). These conclusions, embodied in the few pages which make up the first four papers are amplified somewhat, and supported by observations, in the fifth: (A) layers (in the schist) containing opal or chalcedony are found between layers of unbleached rock consisting of about 75 per cent diatom frustules; every gradation is present between the unbleached rock and that in which the silica has been largely dissolved and redeposited as menilite (apparently nodules of opal, chalcedony, and chert); (B) the solution and deposition took place under marine conditions, because some of the menilite is replaced by glauconite; (C) glauconitization (which proceeds simultaneously with the generation of this mineral) can be seen in the following stages: first, as little greenish patches or clouds on the surface of siliceous cement, then as a cryptocrystalline green covering on calcareous or siliceous particles, and finally as replacement and transformation of calcareous or siliceous grains into material having the optical properties of glauconite. The mineral originates without the presence of organisms, and is formed in the neritic zone where there is a rich supply of silica, potassium, iron, and also an oxidizing environment. Sapropel must not be present.

FILIPESCO, G., Contribution a l'étude micrographique du Flysch des Carpathes roumaines (Gres de Kliwa). C. R. S. de la Soc. Géol. de France, no. 4, pp. 52–54, 1932.

FILIPESCO, M. G., Étude microscopique du sable silicieux de Kliwa et considérations sur son origine et les conditions de sédimentation. Bull. de la Section Scien., Acad. Roumaine, XV-ème Année, No. 7–8, 1932. (6 pages).

After preliminary treatment with acid, washing, and drying, this weakly cemented sandstone considered in the papers was analyzed mechanically with sieves of 2.157, 1.000, 0.631, and 0.263

mm. aperture (nos. 10, 25, 45, and 100). The figures obtained are plotted by use of a simple frequency curve (not cumulative), in which the diameters (or sieve numbers) are plotted against the weight in grams of material retained on each size. The resulting curve, which consists of several straight lines, gives the same information as does a histogram. (Since 95 per cent of the sample was found to consist of grains smaller than 0.263 mm. it seems that the sieves employed gave comparatively little information about the size distribution.)

Separation with bromoform yielded a very small crop of the usual heavy minerals. The following points are worth noting: kyanite shows signs of rounding, regarded as proof of eolian action; zircons have many liquid inclusions, some in the form of negative crystals; tourmaline is generally brown, but indicolite and rubellite are found. Frequencies are indicated by both the symbols and numbers given by Milner (2nd. ed., p. 386).

From the physical character of the grains it is assumed that the greater part of them have been transported by marine agencies, but that a small percentage has been carried and rounded by the wind. Final deposition was in a marine environment. The heavy minerals and rock fragments show derivation from metamorphic terranes, and the requisite area is said to be close at hand.

FILIPESCO, M. G., Un essai de classification des roches sédimentaires. Buletinul Labor. de Miner. al Univ. din Bucuresti, vol. I, 1934 (3 pages).

The important part of this paper is the large table at the end. In it all sediments are arranged fundamentally with respect to two factors: (a) chemical nature of the material deposited, and (b) the factors which have contributed to the accumulation of the material. There are a large number of subdivisions under each of these heads. The complete table is probably comprehensive, but it is the sort of thing which may be done in many ways.

Considering all the papers reviewed, two facts stand out: first, that there is almost no reference to foreign literature, and none to literature in the English language other than to Milner and to a few papers not really on sedimentary topics. Cayeux's methods are followed almost to the exclusion of others. Second, little proof or observation is given in support of many statements. For this reason, it is difficult to tell when the author is giving the results of his own work, or when he is taking the results from others. *If* Filipescu's work is typical of sedimentary work in Rumania, a necessity is seen for closer cooperation in the use of the same units of measurement (sieves, for example), methods of work, and in the interchange of ideas.

LINCOLN DRYDEN
Bryn Mawr College

PALMQVIST, SVEN. *Geochemical Studies on the Iron-Bearing Liassic Series in Southern Sweden*, Meddelanden från Lunds Geologisk-Mineralogiska Institution; No. 60, 1935. 204 pp., 31 tables, 14 figs., 60 chemical analyses of the iron-bearing rocks.

This paper gives the results obtained from a chemical study of the iron-bearing Liassic rocks of southern Sweden, in the region around Kurremolla. This series, consisting of sandstone, oolitic and sideritic ores, clay and coal, strikes in a general N. 45 W. direction and dips steeply to the S.W. The Liassic series is bounded on the N.E. by Silurian and the S.W. by Chalk.

The iron-bearing horizons are divided into three types; ilmenite-rich oolitic ores, sand-rich oolitic ores, and siderite ores. The oolitic ores are composed of chamositic minerals in well developed oolitics with carbonates, ilmenite, and sand. The siderite ores are usually present as independent layers in the sandstone not associated with the oolitic ores and distinctly separated from the surrounding sandstone.

These iron-bearing rocks were found,

by chemical methods, to contain kaolin, ilmenite, apatite, pyrite, siderite, calcium and magnesium carbonates, iron oxides, carbon, sand, and chamositic minerals. The paper includes a description of the general chemical methods used, and describes the identification of some rare elements by spectrographic means. These methods disclosed the presence of boron, germanium, vanadium, zinc, nickel, cobalt, chromium, molybdenum, and arsenic.

The rocks are believed to be shallow marine deposits and may be lagoonal. The presence of fossil plants and clay beds suggests lagoonal conditions. If these are lagoonal deposits they were connected with the sea at all times as shown by the constant percentage of boron present.

Palmqvist correlates the three types of ores with variations in the conditions of deposition. The high ilmenite ores occur in thin strata and are thought to have been deposited in periods of agitated water with the removal of the sand. The sand rich ores are believed to be indicative of less active water which permitted the sand to be deposited. The sand-free siderite ores may indicate periods of tranquil water which allowed the sand-free beds to form. The author believes that oolites indicate agitated conditions.

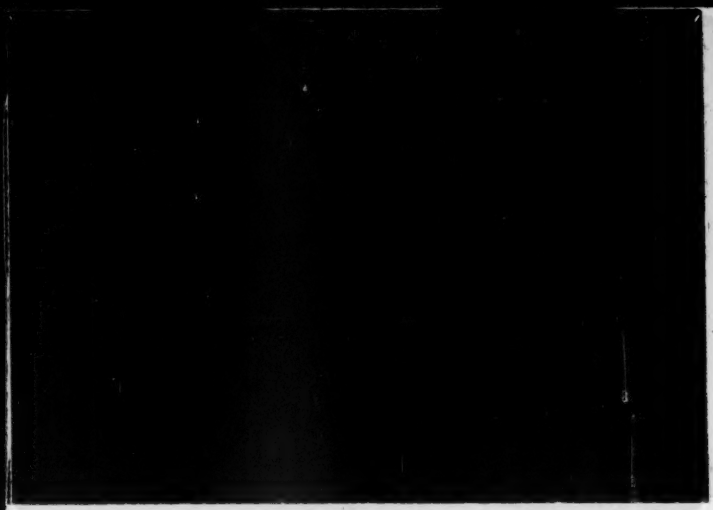
The author attributes the chemical difference of the ores to different modes of origin. The silicate ores are thought to have formed by colloidal flocculation processes while the siderite formed by precipitation from a molecule-dispersive phase. This is supported by the concentration of rare elements in the silicate ores, caused by the greater enrichment properties of colloidal flocculates.

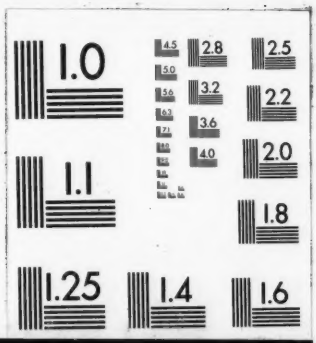
The character of the minerals present point to a primary formation of the different minerals. In no case were any metasomatic alterations observed. All factors favor a primary sedimentary deposition.

There is evidence of slight changes in the environment of deposition from reducing to slightly oxidizing conditions as illustrated by the sporadic appearance of lenses high in ferric iron. The first beds deposited are low in the trivalent iron. There is an increase as the upper horizons are approached, which indicates a progressive change in environment to conditions similar to those prevailing in the present oceans.

RALPH W. MARSDEN

University of Wisconsin

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